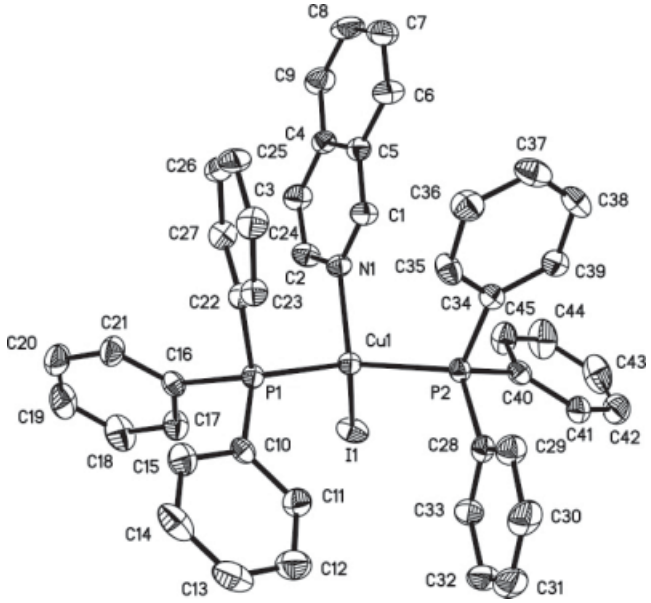
Crystal structure of bis(triphenylphospine-κ*P*)(iodido)-(isoquinoline-κ*N*) copper(I), $[CuI(PC_{18}H_{15})_2(i\text{-}C_9H_7N)]$, $C_{45}H_{37}CuINP_2$

Jian-Bao Li[I], Bin Liu[I], Li-Li Zhou[II], Man-Hua Wu[I], Qiong-Hua Jin[*, I] and Cun-Ling Zhang[III]

[I] Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China
[II] Research Center for Import-Export Chemicals Safety of General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China (AQSIQ), Chinese Academy of Inspection and Quarantine, Beijing 100123, P. R. China
[III] Key Laboratory of Terahertz Optoelectronics, Ministry of Education, Capital Normal University, Beijing 100048, P. R. China

Received December 26, 2011, accepted April 22, 2013, available online April 26, 2013, CCDC no. 1267/3960



Abstract

$C_{45}H_{37}CuINP_2$, monoclinic, $P2_1/n$ (no. 14), a = 10.0738(9) Å, b = 36.195(3) Å, c = 11.564(1) Å, β = 115.047(2)°, V = 3820.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.0461, $wR_{ref}(F^2)$ = 0.0944, T = 298 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.32×0.39×0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.96 cm^{-1}
Diffractometer, scan mode:	Brucker SMART CCD, φ and ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	19005, 6696
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4674
$N(param)_{refined}$:	451
Programs:	SHELX [8]

Source of material

The title compound was prepared by the reaction of CuI, PPh_3 and isoquinoline in the molar ratio of 3:3:1 in a CH_3OH/CH_2Cl_2 solution at the room temperature for 4h. A yellow precipitate was formed and filtered. Yellow crystals were obtained by slow evaporation.

Discussion

Cu(I) complexes of mixed ligand phosphines and heterocyclic nitrogen ligands have been studied because of their diverse structural chemistry and their photophysics properties [1, 2]. We have focused our work on the synthesis of Cu(I) complexes containing triphenylphosphine (PPh_3) and heterocyclic nitrogen ligands for a long time. We have obtained a series of compounds, such as $[CuI(PPh_3)(C_9H_7N)]_2$ [3], $[CuBr(PPh_3)(C_9H_7N)]_2$ [4] and $[CuI(PPh_3)(C_{12}H_8N_2)]$ [5]. Very recently we synthesized two Cu(I) compounds $[CuBr(PPh_3)_2(i\text{-}C_9H_7N)]$ [6] and $[CuCl(PPh_3)(i\text{-}C_9H_7N)]_2$ [7], which contain isoquinoline and PPh_3 at the same time. In the previous paper [7], we have mentioned that there are at least two series of Cu(I) complexes containing isoquinoline and PPh_3, i.e. mononuclear and dinuclear complexes. Here we report a new mononuclear Cu(I) complex $[CuI(PPh_3)_2(i\text{-}C_9H_7N)]$ which has a structure as $[CuBr(PPh_3)_2(i\text{-}C_9H_7N)]$ [6]. The title structure reveals a distorted tetrahedral coordination of the Cu atom, with the four vertices occupied by two P atoms from PPh_3, one N atom from i-C_9H_7N and one I atom. In the title structure, the bond lengths Cu–P (2.2813(13)-2.2935(13) Å) and Cu–N (2.132(4) Å) are very similar to the corresponding distances in $[CuBr(PPh_3)_2(i\text{-}C_9H_7N)]$ [6] (d(Cu–P) = 2.279(1)-2.290(1) Å, d(Cu–N) = 2.135(4) Å), but are longer than those in $[CuI(PPh_3)(C_9H_7N)]_2$ [3] (d(Cu–P) = 2.1945(8) Å, d(Cu–N) = 2.066(2) Å) and in $[CuBr(PPh_3)(C_9H_7N)]_2$ [4] (d(Cu–P) = 2.1997(9) Å and d(Cu–N) = 2.111(3) Å). The Cu–I bond length (2.6288(7) Å) in the title compound is longer than that in [5] (2.6157(6) Å). The different distances in the structure described in [3-6] may be due to the different anions or heterocyclic nitrogen ligands. The bond angles N–Cu–P (102.54(11) and 105.48(11)°) in the title compound are similar to those in $[CuBr(PPh_3)_2(i\text{-}C_9H_7N)]$ [6] (101.5(1) and 105.3(1)°), and smaller than the value found in the similar compound $[CuI(PPh_3)(C_{12}H_8N_2)]$ [5] (110.68(7)°). While two bond angles P–Cu–I (102.07(11) and 116.15(4)°) do not agree with the corresponding bond angle in $[CuI(PPh_3)(C_{12}H_8N_2)]$ [5] (112.70(3)°).

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.4826	0.0967	0.7754	0.051
H(2)	4*e*	0.6038	0.1582	0.5635	0.056
H(3)	4*e*	0.8426	0.1503	0.6939	0.064
H(6)	4*e*	0.6623	0.0657	0.9683	0.068
H(7)	4*e*	0.8994	0.0534	1.0981	0.075
H(8)	4*e*	1.0885	0.0797	1.0629	0.081
H(9)	4*e*	1.0374	0.1177	0.8910	0.076
H(11)	4*e*	–0.0350	0.1649	0.4486	0.060
H(12)	4*e*	–0.2770	0.1811	0.3918	0.082
H(13)	4*e*	–0.3315	0.2300	0.4893	0.083
H(14)	4*e*	–0.1466	0.2620	0.6493	0.080

* Correspondence author (e-mail: jinqh@mail.cnu.edu.cn)

Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(15)	4*e*	0.0956	0.2464	0.7061	0.065
H(17)	4*e*	0.2710	0.2231	0.4146	0.063
H(18)	4*e*	0.3634	0.2783	0.3747	0.085
H(19)	4*e*	0.4760	0.3196	0.5356	0.083
H(20)	4*e*	0.4933	0.3082	0.7364	0.078
H(21)	4*e*	0.3970	0.2547	0.7766	0.063
H(23)	4*e*	0.1388	0.1761	0.8181	0.047
H(24)	4*e*	0.2489	0.1607	1.0314	0.061
H(25)	4*e*	0.4992	0.1610	1.1429	0.065
H(26)	4*e*	0.6436	0.1764	1.0390	0.060
H(27)	4*e*	0.5356	0.1899	0.8234	0.054
H(29)	4*e*	–0.0626	0.0634	0.5951	0.062
H(30)	4*e*	–0.3127	0.0709	0.4982	0.078

Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(31)	4*e*	–0.4272	0.0957	0.2986	0.080
H(32)	4*e*	–0.2898	0.1122	0.1888	0.075
H(33)	4*e*	–0.0363	0.1071	0.2865	0.058
H(35)	4*e*	0.2114	0.1024	0.7684	0.057
H(36)	4*e*	0.2887	0.0756	0.9674	0.067
H(37)	4*e*	0.3433	0.0136	0.9908	0.075
H(38)	4*e*	0.3267	–0.0211	0.8191	0.078
H(39)	4*e*	0.2494	0.0059	0.6211	0.061
H(41)	4*e*	0.0178	0.0222	0.3453	0.060
H(42)	4*e*	0.0756	–0.0190	0.2227	0.079
H(43)	4*e*	0.3045	–0.0185	0.2245	0.096
H(44)	4*e*	0.4792	0.0227	0.3530	0.096
H(45)	4*e*	0.4217	0.0644	0.4727	0.071

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	4*e*	0.29347(6)	0.13763(2)	0.53644(5)	0.0349(3)	0.0366(3)	0.0318(3)	0.0001(3)	0.0159(3)	0.0018(3)
P(1)	4*e*	0.2458(1)	0.18965(3)	0.6237(1)	0.0392(7)	0.0314(7)	0.0295(7)	–0.0025(5)	0.0167(6)	0.0007(5)
P(2)	4*e*	0.1777(1)	0.08213(3)	0.5225(1)	0.0346(7)	0.0295(6)	0.0393(7)	0.0005(5)	0.0176(6)	–0.0013(6)
I(1)	4*e*	0.29608(4)	0.14548(1)	0.31142(3)	0.0655(3)	0.0709(3)	0.0345(2)	0.0190(2)	0.0287(2)	0.0114(2)
N(1)	4*e*	0.5189(4)	0.1277(1)	0.6566(4)	0.032(2)	0.045(2)	0.036(2)	0.000(2)	0.013(2)	0.002(2)
C(1)	4*e*	0.5571(5)	0.1070(1)	0.7585(5)	0.034(3)	0.053(3)	0.040(3)	–0.003(2)	0.015(2)	0.010(3)
C(2)	4*e*	0.6287(5)	0.1433(1)	0.6352(5)	0.044(3)	0.056(3)	0.046(3)	0.003(3)	0.023(3)	0.013(3)
C(3)	4*e*	0.7718(6)	0.1385(2)	0.7122(5)	0.038(3)	0.066(4)	0.058(4)	–0.006(3)	0.022(3)	0.006(3)
C(4)	4*e*	0.8145(5)	0.1155(1)	0.8205(5)	0.033(3)	0.045(3)	0.043(3)	0.006(2)	0.014(2)	–0.004(3)
C(5)	4*e*	0.7019(5)	0.0993(1)	0.8441(5)	0.039(3)	0.042(3)	0.037(3)	0.003(2)	0.016(2)	–0.002(2)
C(6)	4*e*	0.7367(6)	0.0761(2)	0.9513(5)	0.048(4)	0.071(4)	0.041(3)	0.002(3)	0.010(3)	0.005(3)
C(7)	4*e*	0.8775(7)	0.0691(2)	1.0286(6)	0.066(4)	0.066(4)	0.047(4)	0.010(3)	0.015(3)	0.007(3)
C(8)	4*e*	0.9917(7)	0.0847(2)	1.0074(6)	0.044(4)	0.085(5)	0.050(4)	0.017(3)	–0.002(3)	–0.006(4)
C(9)	4*e*	0.9611(6)	0.1074(2)	0.9052(6)	0.037(3)	0.075(4)	0.068(4)	0.000(3)	0.013(3)	–0.002(4)
C(10)	4*e*	0.0563(5)	0.2039(1)	0.5840(5)	0.044(3)	0.031(3)	0.039(3)	0.002(2)	0.021(2)	0.011(2)
C(11)	4*e*	–0.0568(6)	0.1846(2)	0.4894(5)	0.051(4)	0.049(3)	0.048(3)	–0.002(3)	0.018(3)	0.001(3)
C(12)	4*e*	–0.2021(7)	0.1943(2)	0.4548(6)	0.049(4)	0.071(4)	0.074(5)	0.002(3)	0.014(3)	0.011(4)
C(13)	4*e*	–0.2343(7)	0.2232(2)	0.5134(7)	0.049(4)	0.073(5)	0.088(5)	0.016(3)	0.030(4)	0.037(4)
C(14)	4*e*	–0.1237(7)	0.2426(2)	0.6084(7)	0.070(5)	0.051(4)	0.094(5)	0.023(3)	0.050(4)	0.022(4)
C(15)	4*e*	0.0214(6)	0.2330(2)	0.6429(5)	0.060(4)	0.048(3)	0.058(4)	0.003(3)	0.029(3)	–0.004(3)
C(16)	4*e*	0.3260(5)	0.2329(1)	0.6008(5)	0.045(3)	0.032(3)	0.041(3)	0.002(2)	0.023(2)	0.006(2)
C(17)	4*e*	0.3153(6)	0.2401(1)	0.4800(5)	0.071(4)	0.045(3)	0.049(3)	–0.005(3)	0.032(3)	0.000(3)
C(18)	4*e*	0.3714(7)	0.2732(2)	0.4562(7)	0.103(5)	0.058(4)	0.073(5)	–0.003(4)	0.059(4)	0.020(4)
C(19)	4*e*	0.4375(7)	0.2979(2)	0.5518(7)	0.075(4)	0.045(4)	0.102(6)	–0.016(3)	0.050(4)	0.008(4)
C(20)	4*e*	0.4480(6)	0.2912(2)	0.6713(6)	0.072(4)	0.047(4)	0.065(4)	–0.019(3)	0.020(4)	–0.004(3)
C(21)	4*e*	0.3913(6)	0.2590(1)	0.6953(5)	0.061(4)	0.042(3)	0.052(4)	–0.014(3)	0.023(3)	–0.003(3)
C(22)	4*e*	0.3259(5)	0.1837(1)	0.7971(4)	0.044(3)	0.034(3)	0.030(3)	–0.005(2)	0.019(2)	0.000(2)
C(23)	4*e*	0.2404(5)	0.1756(1)	0.8612(5)	0.042(3)	0.041(3)	0.037(3)	0.000(2)	0.020(2)	–0.001(2)
C(24)	4*e*	0.3069(6)	0.1668(2)	0.9895(5)	0.069(4)	0.058(3)	0.035(3)	–0.006(3)	0.031(3)	0.000(3)
C(25)	4*e*	0.4563(6)	0.1669(2)	1.0566(5)	0.063(4)	0.063(4)	0.025(3)	0.005(3)	0.008(3)	0.004(3)
C(26)	4*e*	0.5421(6)	0.1759(1)	0.9944(5)	0.044(3)	0.057(4)	0.040(3)	0.000(3)	0.009(3)	–0.004(3)
C(27)	4*e*	0.4771(6)	0.1841(1)	0.8651(5)	0.046(3)	0.051(3)	0.044(3)	–0.003(3)	0.026(3)	–0.001(3)
C(28)	4*e*	–0.0220(5)	0.0841(1)	0.4514(5)	0.041(3)	0.035(3)	0.045(3)	–0.003(2)	0.020(3)	–0.006(2)
C(29)	4*e*	–0.1069(6)	0.0735(2)	0.5137(5)	0.043(3)	0.057(4)	0.056(4)	–0.002(3)	0.023(3)	0.000(3)
C(30)	4*e*	–0.2572(6)	0.0780(2)	0.4550(7)	0.036(4)	0.073(4)	0.092(5)	–0.007(3)	0.033(4)	–0.010(4)
C(31)	4*e*	–0.3261(6)	0.0927(2)	0.3360(7)	0.033(3)	0.069(4)	0.088(5)	0.009(3)	0.017(4)	–0.013(4)
C(32)	4*e*	–0.2436(6)	0.1029(2)	0.2713(6)	0.045(4)	0.063(4)	0.063(4)	0.014(3)	0.007(3)	0.000(3)
C(33)	4*e*	–0.0915(6)	0.0993(1)	0.3292(5)	0.041(3)	0.051(3)	0.052(4)	0.006(3)	0.019(3)	0.002(3)
C(34)	4*e*	0.2255(5)	0.0575(1)	0.6734(5)	0.029(3)	0.040(3)	0.043(3)	0.001(2)	0.021(2)	0.005(2)
C(35)	4*e*	0.2357(6)	0.0774(1)	0.7782(5)	0.070(4)	0.038(3)	0.045(3)	0.005(3)	0.036(3)	0.005(3)
C(36)	4*e*	0.2808(6)	0.0615(2)	0.8976(5)	0.067(4)	0.063(4)	0.042(3)	0.002(3)	0.026(3)	0.001(3)
C(37)	4*e*	0.3136(6)	0.0246(2)	0.9112(6)	0.063(4)	0.074(4)	0.051(4)	0.011(3)	0.025(3)	0.024(3)
C(38)	4*e*	0.3033(7)	0.0040(2)	0.8089(6)	0.078(4)	0.044(3)	0.067(4)	0.009(3)	0.026(4)	0.020(3)
C(39)	4*e*	0.2581(6)	0.0202(1)	0.6906(5)	0.052(3)	0.045(3)	0.047(4)	0.003(3)	0.013(3)	0.000(3)
C(40)	4*e*	0.2146(5)	0.0479(1)	0.4240(5)	0.040(3)	0.036(3)	0.045(3)	0.006(2)	0.021(2)	0.003(2)
C(41)	4*e*	0.1109(6)	0.0225(1)	0.3470(5)	0.057(4)	0.039(3)	0.049(3)	0.001(3)	0.017(3)	–0.004(3)
C(42)	4*e*	0.1454(8)	–0.0021(2)	0.2736(6)	0.088(5)	0.054(4)	0.048(4)	–0.002(3)	0.021(4)	–0.011(3)
C(43)	4*e*	0.2820(9)	–0.0019(2)	0.2750(7)	0.125(7)	0.050(4)	0.090(6)	0.012(4)	0.069(5)	–0.014(4)
C(44)	4*e*	0.3858(8)	0.0228(2)	0.3508(7)	0.084(5)	0.062(4)	0.123(6)	0.005(4)	0.073(5)	–0.023(4)
C(45)	4*e*	0.3514(6)	0.0475(2)	0.4229(6)	0.054(4)	0.050(3)	0.084(5)	–0.002(3)	0.039(3)	–0.015(3)

Acknowledgments. This work has been supported by the National Science Foundation of China (grant no. 21171119), the Project-sponsored by SRF for ROCS and SEM, the National High Technology Research and Development Program 863 of China (grant No. 2012AA063201) and the Committee of Education of the Beijing Foundation of China (grant No. KM201210028020).

References

1. Xiao, Y. L.; Jin, Q. H.; Deng, Y. H.; Li, Z. F.; Yang, W.; Wu, M. H.; Zhang, C. L.: Synthesis and structural characterization of five copper(I) complexes of [2,3-*f*]-pyrazino-[1,10]phenanthroline-2,3-dicarbonitrile and triphenylphosphine. Inorg. Chem. Commun. **15** (2012)146-150.
2. Jin, Q. H.; Chen, L. M.; Yang, L.; Li, P. Z.: The synthesis, structure and luminescence of two 1D pyrazine-connected Cu(I)-dppm polymers. Inorg. Chim. Acta **362** (2009) 1743-1748.
3. Jin, Q. H.; Xin, X. Q.: Di-μ-iodo-1:2κ^4*I*-bis(quinoline)-1κ*N*,2κ*N*-bis(triphenylphosphine)-1κ*P*,2κ*P*-dicopper(I). Acta Crystallogr. **C55** (1999) 341-343.
4. Jin, Q. H.; Long, D. L.; Wang, Y. X.; Xin, X. Q.: Di-μ-bromo-1:2κ^4*Br*-bis(quinoline)-1κ*N*,2κ*N*-bis(triphenylphosphine)-1κ*P*,2κ*P*-dicopper(I). Acta Crystallogr. **C54** (1998) 948-949.
5. Jin, Q. H.; Xin, X. L.; Dong, C. J.; Zhu, H. J.: Iodo(1,10-phenanthroline-*N,N'*)(triphenylphosphine)copper(I). Acta Crystallogr. **C54** (1998) 1087-1089.
6. Li, J. B.; Wu, M. H.; Jiang, Y. H.; Wen, J.; Jin, Q. H.; Gong, H. L.: Crystal structure of bis(triphenylphospine-κ*P*)(bromo-κ*Br*)-(isoquinoline-κ*N*)copper(I), $CuBr(PC_{18}H_{15})_2(i\text{-}C_9H_7N)$. Z. Kristallogr. NCS **226** (2011) 109-111.
7. Li, J. B.; Xiao, Y. L.; Wu, M. H.; Jiang, Y. H.; Jin, Q. H.; Zhang C. L.: Crystal structure of bis(triphenylphospine-κ*P*)bis(μ-chloro-κ*Cl*) bis(isoquinoline-κ*N*)copper(I), $[CuCl(PC_{18}H_{15})(i\text{-}C_9H_7N)]_2$. Z. Kristallogr. NCS **226** (2011) 97-98.
8. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.